

Supplementary Information

Electrochemical Impedance Spectroscopy

Recent work by Teran et al¹ demonstrated a correlation between the ionic conductivity and morphology in block copolymer salt mixtures. Their work revealed a discontinuous increase in ionic conductivity as the sample transitioned from ordered lamellae to a disordered morphology. This work sought to gain insight into the ion transport behavior within coexistence window of the ODT. The temperature dependent ionic conductivity values of the three SEO(1.7-1.4)/LiTfI($r=0.075$) samples measured in this study are shown in Figure S1, where the coexistence window determined through SAXS is highlighted in yellow. All three samples demonstrate a discontinuous increase in ionic conductivity between the ordered lamellar and disordered phases present at low and high temperatures, respectively. The ionic conductivity within the coexistence temperature window is unremarkable and monotonically increases from the lower lamellar phase conductivity to the higher disordered phase conductivity.

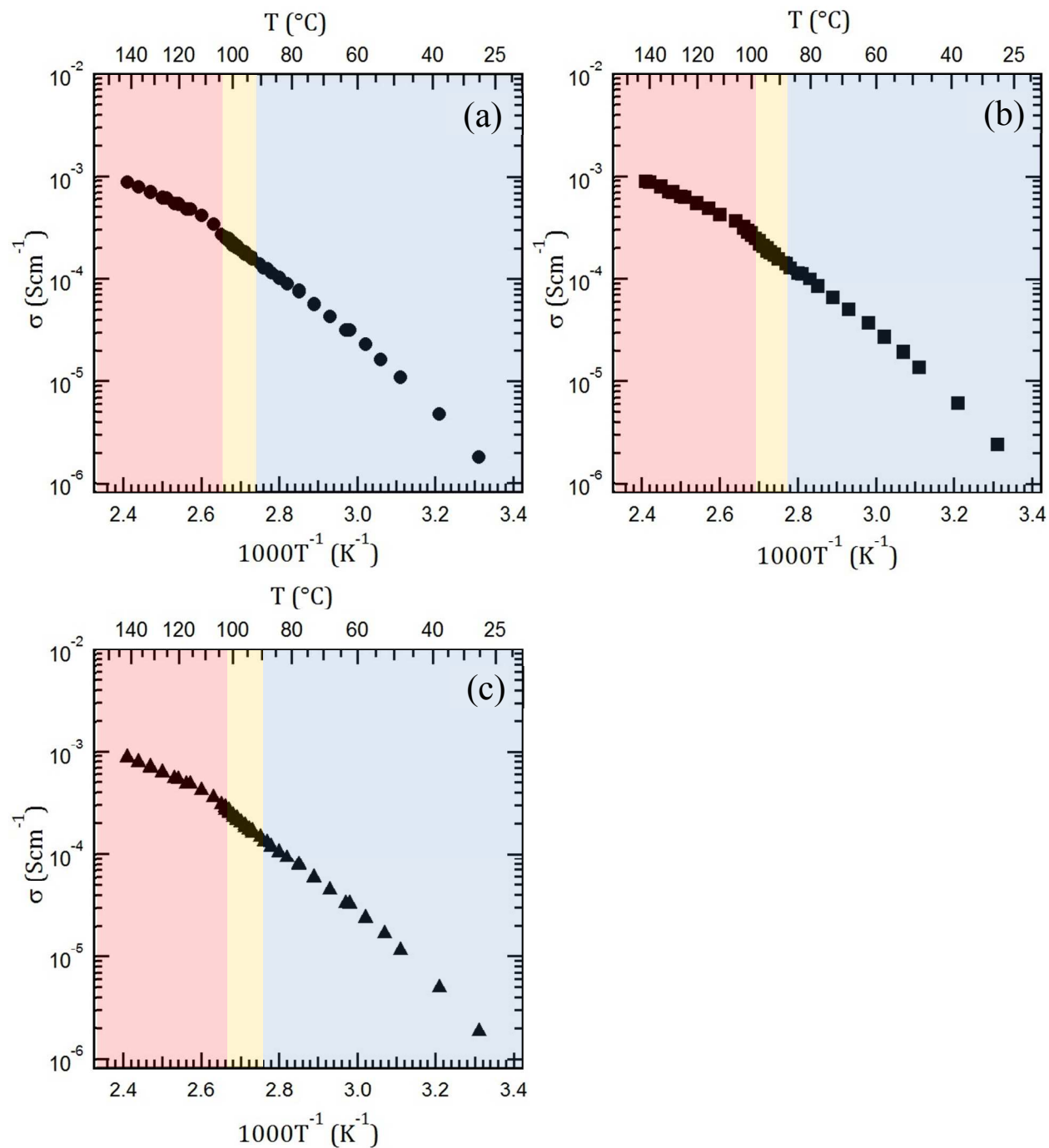


Figure S1. Ionic conductivity for SEO(1.7-1.4)/LiTFSI($r=0.075$) measured *in situ* during SAXS for (a) Sample 1, (b) Sample 2, and (c) Sample 3. Regions shaded in blue indicate temperatures below the ODT, regions in yellow encompass the occurrence of an ODT, and regions in red correspond to a fully disordered polymer melt.

SAXS Profile Fitting

The absolute SAXS intensity profiles obtained for the SEO(1.7-1.4)/LiTFSI($r=0.075$) samples at all temperatures were fit with the functions described in equations 1-13 of the main text. Table S1 provides the fit parameters for the curve shown as an example in Figure 1 of the main text. The fit parameters for Sample 1 at the remaining temperatures are organized into Tables S2-4. Table S2 provides I_{ord} fit parameters, Table S3 provides I_{dis} fit parameters, and Table S4 provides I_{bgd} fit parameters. For consistency with the recent work by Teran et al², the PDI for all I_{dis} fitting was set to PDI = 1.035.

The SAXS profile fit parameters were used to calculate physically relevant quantities through Equations 14-19 of the main text. These values are summarized in Table S5 for Sample 1 at all temperatures measured.

Table S1. Fit parameters for the curve shown in Figure 1 of the main text. The measured sample temperature was 97.2 ± 1.5 °C. All reported errors represent one standard deviation of the fitting error.

Parameter	Value	(\pm)	Units
y_0	28.3	(0.7)	[cm ⁻¹]
q^*	0.82471	(9E-05)	[nm ⁻¹]
w	6.0E-03	(7.7E-05)	[nm ⁻¹]
f	0.499	-	[-]
N	49.2	-	[sites chain ⁻¹]
C	2.58E-02	(1.4E-04)	[cm ⁻¹]
R_g	2.306	(1E-03)	[nm]
χ_{eff}	0.2049	(3.7E-05)	[-]
y_1	3.76E-02	(3.E-04)	[cm ⁻¹]
y_2	0.54	(0.01)	[nm ⁻¹]

Table S2. Sample 1 fit parameters for I_{ord} at all measured temperatures. T_{set} represents the controller setpoint, $Time_{meas}$ is the cumulative experimental time since the first scattering measurement, T_{meas} is the experimentally measured temperature, and y_0 , q^* , w correspond to the constants in Equation 2 of the main text. All reported errors represent one standard deviation of the fitting error.

T_{set} [°C]	$Time_{meas}$ [hr:min]	T_{meas} (±) [°C]	y_0 (±) [cm ⁻¹]	q^* (±) [nm ⁻¹]	w (±) [nm ⁻¹]
30	0:00	28.9 (0.1)	86.5 (1.8)	0.82109 (4E-05)	5.6E-03 (3.2E-05)
40	0:42	38.6 (0.3)	84.1 (1.8)	0.82213 (4E-05)	5.5E-03 (3.3E-05)
	0:52	38.6 (0.3)	83.2 (1.7)	0.82203 (4E-05)	5.5E-03 (3.3E-05)
	1:05	38.6 (0.3)	83.5 (1.8)	0.82202 (4E-05)	5.5E-03 (3.3E-05)
50	1:21	48.3 (0.5)	79.9 (1.7)	0.82318 (4E-05)	5.5E-03 (3.4E-05)
	1:31	48.3 (0.5)	79.2 (1.7)	0.82301 (4E-05)	5.5E-03 (3.4E-05)
	1:44	48.3 (0.5)	76.7 (1.6)	0.82352 (4E-05)	5.6E-03 (3.4E-05)
55	1:59	53.2 (0.6)	75.1 (1.6)	0.82405 (4E-05)	5.6E-03 (3.4E-05)
	2:09	53.2 (0.6)	74.4 (1.6)	0.82394 (4E-05)	5.5E-03 (3.4E-05)
	2:18	53.2 (0.5)	74.4 (1.6)	0.82387 (4E-05)	5.5E-03 (3.4E-05)
60	2:35	58.1 (0.7)	72.5 (1.5)	0.82439 (5E-05)	5.5E-03 (3.4E-05)
	2:44	58.1 (0.7)	71.8 (1.5)	0.82429 (5E-05)	5.5E-03 (3.4E-05)
	2:54	58.2 (0.7)	71.8 (1.5)	0.82419 (5E-05)	5.5E-03 (3.4E-05)
65	3:15	63.0 (0.8)	71.1 (1.5)	0.82468 (5E-05)	5.5E-03 (3.4E-05)
	3:24	63.0 (0.7)	70.9 (1.5)	0.82457 (5E-05)	5.5E-03 (3.4E-05)
	3:37	63.0 (0.7)	71.3 (1.5)	0.82446 (5E-05)	5.5E-03 (3.4E-05)
70	3:52	67.9 (0.9)	70.4 (1.5)	0.82492 (5E-05)	5.5E-03 (3.4E-05)
	4:02	67.9 (0.9)	70.7 (1.5)	0.82480 (5E-05)	5.6E-03 (3.4E-05)
	4:12	67.9 (0.8)	71.6 (1.5)	0.82465 (5E-05)	5.6E-03 (3.4E-05)
75	4:28	72.8 (1.0)	72.3 (1.5)	0.82563 (5E-05)	5.6E-03 (3.5E-05)
	4:39	72.7 (0.9)	72.5 (1.5)	0.82549 (5E-05)	5.6E-03 (3.5E-05)
	4:49	72.8 (0.9)	72.6 (1.5)	0.82537 (5E-05)	5.6E-03 (3.5E-05)
80	5:12	77.7 (1.1)	71.6 (1.5)	0.82586 (5E-05)	5.6E-03 (3.5E-05)
	5:20	77.8 (1.1)	70.9 (1.5)	0.82575 (5E-05)	5.6E-03 (3.5E-05)
	5:25	77.9 (1.1)	70.7 (1.5)	0.82571 (5E-05)	5.6E-03 (3.5E-05)
	5:35	78.3 (1.1)	71.1 (1.5)	0.82562 (5E-05)	5.6E-03 (3.5E-05)
83	5:52	81.1 (1.1)	69.1 (1.4)	0.82608 (5E-05)	5.7E-03 (3.6E-05)
	6:02	81.1 (1.1)	69.1 (1.4)	0.82597 (5E-05)	5.7E-03 (3.6E-05)
	6:12	81.0 (1.1)	69.0 (1.4)	0.82584 (5E-05)	5.7E-03 (3.6E-05)
86	6:28	83.9 (1.2)	68.4 (1.4)	0.82609 (5E-05)	5.7E-03 (3.7E-05)
	6:37	83.7 (1.2)	67.7 (1.4)	0.82582 (5E-05)	5.7E-03 (3.6E-05)
	6:48	83.6 (1.2)	67.8 (1.4)	0.82570 (5E-05)	5.7E-03 (3.6E-05)
89	7:04	86.5 (1.2)	67.3 (1.4)	0.82596 (5E-05)	5.7E-03 (3.7E-05)
	7:14	86.5 (1.2)	67.1 (1.4)	0.82586 (5E-05)	5.7E-03 (3.7E-05)
	7:24	86.4 (1.2)	67.1 (1.4)	0.82574 (5E-05)	5.7E-03 (3.7E-05)
	7:34	86.4 (1.2)	67.2 (1.4)	0.82567 (5E-05)	5.7E-03 (3.7E-05)

Table S2 (Continued).

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (\pm) [°C]	y_0 (\pm) [cm ⁻¹]	q^* (\pm) [nm ⁻¹]	w (\pm) [nm ⁻¹]
91	7:49	88.4 (1.3)	66.5 (1.4)	0.82580 (5E-05)	5.7E-03 (3.7E-05)
	7:59	88.5 (1.3)	66.4 (1.4)	0.82570 (5E-05)	5.7E-03 (3.7E-05)
	8:09	88.6 (1.3)	66.3 (1.4)	0.82565 (5E-05)	5.7E-03 (3.7E-05)
	8:19	88.7 (1.3)	66.2 (1.4)	0.82557 (5E-05)	5.7E-03 (3.7E-05)
93	8:36	90.7 (1.3)	65.6 (1.4)	0.82574 (5E-05)	5.7E-03 (3.8E-05)
	8:46	90.7 (1.3)	65.1 (1.4)	0.82561 (5E-05)	5.7E-03 (3.7E-05)
	8:56	90.7 (1.3)	65.2 (1.4)	0.82557 (5E-05)	5.7E-03 (3.8E-05)
95	9:31	92.8 (1.4)	63.5 (1.3)	0.82582 (5E-05)	5.7E-03 (3.9E-05)
	9:42	92.7 (1.4)	63.3 (1.3)	0.82572 (5E-05)	5.7E-03 (3.9E-05)
	9:51	92.9 (1.4)	62.7 (1.3)	0.82570 (5E-05)	5.7E-03 (3.9E-05)
96	10:06	93.8 (1.4)	61.1 (1.3)	0.82573 (5E-05)	5.8E-03 (4.0E-05)
	10:16	93.8 (1.4)	60.4 (1.3)	0.82560 (5E-05)	5.8E-03 (4.1E-05)
	10:26	93.6 (1.4)	60.3 (1.3)	0.82553 (5E-05)	5.8E-03 (4.1E-05)
97	10:44	94.5 (1.4)	58.0 (1.2)	0.82557 (6E-05)	5.8E-03 (4.3E-05)
	10:54	94.5 (1.4)	57.1 (1.2)	0.82545 (6E-05)	5.8E-03 (4.3E-05)
	11:04	94.4 (1.4)	57.0 (1.2)	0.82542 (6E-05)	5.8E-03 (4.3E-05)
	11:14	94.3 (1.4)	56.8 (1.2)	0.82535 (6E-05)	5.8E-03 (4.4E-05)
	11:24	94.3 (1.4)	56.2 (1.2)	0.82528 (6E-05)	5.8E-03 (4.4E-05)
	11:32	94.3 (1.4)	55.5 (1.2)	0.82520 (6E-05)	5.8E-03 (4.4E-05)
98	11:48	95.3 (1.4)	52.0 (1.1)	0.82525 (6E-05)	5.8E-03 (4.7E-05)
	11:58	95.3 (1.4)	50.4 (1.1)	0.82517 (6E-05)	5.8E-03 (4.8E-05)
	12:08	95.3 (1.4)	49.4 (1.1)	0.82513 (6E-05)	5.8E-03 (4.9E-05)
	12:18	95.3 (1.4)	48.9 (1.1)	0.82510 (6E-05)	5.9E-03 (5.0E-05)
	12:29	95.3 (1.4)	48.2 (1.0)	0.82505 (7E-05)	5.8E-03 (5.0E-05)
	12:38	95.3 (1.4)	47.2 (1.0)	0.82497 (7E-05)	5.9E-03 (5.1E-05)
99	12:54	96.3 (1.4)	42.4 (0.9)	0.82502 (7E-05)	5.9E-03 (5.6E-05)
	13:04	96.2 (1.4)	41.5 (0.9)	0.82499 (7E-05)	5.9E-03 (5.7E-05)
	13:14	96.2 (1.4)	40.7 (0.9)	0.82492 (7E-05)	5.8E-03 (5.8E-05)
	13:24	96.3 (1.4)	39.4 (0.9)	0.82491 (8E-05)	5.9E-03 (5.9E-05)
	13:34	96.2 (1.4)	38.7 (0.9)	0.82487 (8E-05)	5.9E-03 (6.0E-05)
	13:45	96.2 (1.4)	37.6 (0.8)	0.82483 (8E-05)	5.9E-03 (6.1E-05)
100	14:01	97.2 (1.5)	31.9 (0.7)	0.82487 (9E-05)	6.0E-03 (7.0E-05)
	14:11	97.2 (1.5)	31.3 (0.7)	0.82482 (9E-05)	5.9E-03 (7.1E-05)
	14:21	97.2 (1.5)	30.4 (0.7)	0.82480 (9E-05)	6.0E-03 (7.3E-05)
	14:32	97.2 (1.5)	29.4 (0.7)	0.82478 (9E-05)	6.0E-03 (7.4E-05)
	14:42	97.2 (1.5)	28.8 (0.7)	0.82474 (9E-05)	6.0E-03 (7.6E-05)
	14:52	97.2 (1.5)	28.3 (0.7)	0.82471 (9E-05)	6.0E-03 (7.7E-05)

Table S2 (Continued).

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (\pm) [°C]	y_0 (\pm) [cm ⁻¹]	q^* (\pm) [nm ⁻¹]	w (\pm) [nm ⁻¹]
101	15:23	98.2 (1.5)	23.4 (0.6)	0.82490 (1E-04)	6.0E-03 (8.9E-05)
	15:33	98.1 (1.5)	22.1 (0.6)	0.82483 (1E-04)	6.0E-03 (9.2E-05)
	15:41	98.1 (1.5)	21.7 (0.6)	0.82481 (1E-04)	6.0E-03 (9.3E-05)
	15:51	98.2 (1.5)	21.2 (0.6)	0.82481 (1E-04)	6.1E-03 (9.5E-05)
	15:58	98.2 (1.5)	21.0 (0.6)	0.82477 (1E-04)	6.0E-03 (9.6E-05)
102	16:14	99.2 (1.5)	16.8 (0.5)	0.82477 (1E-04)	6.1E-03 (1.1E-04)
	16:24	99.2 (1.5)	16.0 (0.5)	0.82474 (1E-04)	6.0E-03 (1.2E-04)
	16:34	99.2 (1.5)	14.8 (0.4)	0.82471 (1E-04)	6.1E-03 (1.2E-04)
	16:44	99.2 (1.5)	14.6 (0.4)	0.82470 (1E-04)	6.0E-03 (1.3E-04)
	16:54	99.3 (1.5)	14.2 (0.4)	0.82476 (1E-04)	6.2E-03 (1.3E-04)
	17:04	99.3 (1.5)	13.7 (0.4)	0.82474 (2E-04)	6.2E-03 (1.3E-04)
103	17:21	100.3 (1.5)	10.2 (0.4)	0.82484 (2E-04)	6.2E-03 (1.7E-04)
	17:31	100.3 (1.5)	9.0 (0.3)	0.82471 (2E-04)	6.1E-03 (1.8E-04)
	17:41	100.4 (1.5)	8.4 (0.3)	0.82476 (2E-04)	6.1E-03 (1.9E-04)
	17:51	100.3 (1.5)	8.3 (0.3)	0.82482 (2E-04)	6.2E-03 (2.0E-04)
	18:01	100.3 (1.5)	8.5 (0.3)	0.82486 (2E-04)	6.2E-03 (1.9E-04)
104	18:27	101.3 (1.6)	6.8 (0.3)	0.82484 (2E-04)	6.1E-03 (2.3E-04)
	18:37	101.2 (1.6)	6.3 (0.3)	0.82471 (3E-04)	5.9E-03 (2.4E-04)
	18:47	101.2 (1.6)	6.5 (0.3)	0.82488 (3E-04)	6.1E-03 (2.4E-04)
	18:57	101.2 (1.6)	5.9 (0.3)	0.82471 (3E-04)	5.9E-03 (2.5E-04)
	19:07	101.1 (1.6)	6.1 (0.3)	0.82488 (3E-04)	6.0E-03 (2.5E-04)
	19:17	101.2 (1.6)	5.9 (0.3)	0.82481 (3E-04)	6.0E-03 (2.6E-04)
105	19:32	102.1 (1.6)	4.5 (0.3)	0.82481 (3E-04)	5.9E-03 (3.2E-04)
	19:42	102.1 (1.6)	3.5 (0.2)	0.82462 (4E-04)	5.8E-03 (3.9E-04)
	19:52	102.0 (1.6)	3.3 (0.2)	0.82468 (4E-04)	5.7E-03 (4.1E-04)
	20:02	102.0 (1.6)	3.7 (0.2)	0.82489 (4E-04)	5.9E-03 (3.8E-04)
	20:03	101.9 (1.6)	3.7 (0.2)	0.82487 (4E-04)	5.9E-03 (3.7E-04)
	20:13	102.0 (1.6)	3.6 (0.2)	0.82487 (4E-04)	5.9E-03 (3.9E-04)
106	20:30	102.9 (1.6)	2.5 (0.2)	0.82486 (5E-04)	5.8E-03 (5.3E-04)
	20:40	102.9 (1.6)	2.2 (0.2)	0.82497 (6E-04)	5.7E-03 (5.8E-04)
	20:50	102.8 (1.6)	1.4 (0.2)	0.82498 (9E-04)	5.8E-03 (8.9E-04)
	21:17	102.9 (1.6)	1.4 (0.2)	0.82497 (9E-04)	5.7E-03 (9.0E-04)
107	21:43	103.8 (1.6)	1.4 (0.2)	0.82503 (8E-04)	5.3E-03 (8.4E-04)
	21:53	103.8 (1.6)	0.8 (0.2)	0.82494 (1E-03)	4.9E-03 (1.3E-03)
	22:03	103.8 (1.6)	1.1 (0.2)	0.82521 (1E-03)	5.2E-03 (1.0E-03)
	22:13	103.8 (1.6)	1.1 (0.2)	0.82517 (1E-03)	5.2E-03 (1.1E-03)
	22:23	103.8 (1.6)	1.0 (0.2)	0.82524 (1E-03)	5.1E-03 (1.1E-03)
	22:31	103.9 (1.6)	1.0 (0.2)	0.82524 (1E-03)	5.1E-03 (1.1E-03)

Table S3. Sample 1 fit parameters for I_{dis} at all measured temperatures. T_{set} represents the controller setpoint, $Time_{meas}$ is the cumulative experimental time since the first scattering measurement, T_{meas} is the experimentally measured temperature, and f , N , C , R_g , χ_{eff} correspond to the constants in Equations 3-12 of the main text. f and N were calculated based on the temperature dependence of the polymer block densities, as described in ref ².

All reported errors represent one standard deviation of the fitting error.

T_{set} [°C]	$Time_{meas}$ [hr:min]	T_{meas} (±) [°C]	f -	N [sites chain ⁻¹]	C (±) [cm ⁻¹]	R_g (±) [nm]	χ_{eff} (±) -
30	0:00	28.9 (0.1)	0.500	47.2	1.41E-02 (1.4E-04)	2.264 (2E-03)	0.2086 (1.3E-04)
40	0:42	38.6 (0.3)	0.500	47.5	1.50E-02 (1.5E-04)	2.263 (2E-03)	0.2069 (1.3E-04)
	0:52	38.6 (0.3)	0.500	47.5	1.49E-02 (1.5E-04)	2.264 (2E-03)	0.2068 (1.3E-04)
	1:05	38.6 (0.3)	0.500	47.5	1.50E-02 (1.5E-04)	2.264 (2E-03)	0.2069 (1.3E-04)
50	1:21	48.3 (0.5)	0.500	47.8	1.53E-02 (1.6E-04)	2.263 (2E-03)	0.2057 (1.3E-04)
	1:31	48.3 (0.5)	0.500	47.8	1.53E-02 (1.6E-04)	2.263 (2E-03)	0.2056 (1.3E-04)
	1:44	48.3 (0.5)	0.500	47.8	1.51E-02 (1.6E-04)	2.262 (2E-03)	0.2055 (1.4E-04)
55	1:59	53.2 (0.6)	0.500	47.9	1.49E-02 (1.5E-04)	2.261 (2E-03)	0.2053 (1.3E-04)
	2:09	53.2 (0.6)	0.500	47.9	1.47E-02 (1.5E-04)	2.261 (2E-03)	0.2053 (1.3E-04)
	2:18	53.2 (0.5)	0.500	47.9	1.46E-02 (1.5E-04)	2.261 (2E-03)	0.2053 (1.3E-04)
60	2:35	58.1 (0.7)	0.500	48.1	1.46E-02 (1.5E-04)	2.261 (2E-03)	0.2047 (1.3E-04)
	2:44	58.1 (0.7)	0.500	48.1	1.46E-02 (1.5E-04)	2.261 (2E-03)	0.2047 (1.3E-04)
	2:54	58.2 (0.7)	0.500	48.1	1.46E-02 (1.5E-04)	2.262 (2E-03)	0.2047 (1.3E-04)
65	3:15	63.0 (0.8)	0.500	48.2	1.49E-02 (1.5E-04)	2.262 (2E-03)	0.2041 (1.2E-04)
	3:24	63.0 (0.7)	0.500	48.2	1.49E-02 (1.5E-04)	2.262 (2E-03)	0.2041 (1.2E-04)
	3:37	63.0 (0.7)	0.500	48.2	1.50E-02 (1.5E-04)	2.263 (2E-03)	0.2041 (1.2E-04)
70	3:52	67.9 (0.9)	0.500	48.4	1.52E-02 (1.5E-04)	2.262 (2E-03)	0.2036 (1.2E-04)
	4:02	67.9 (0.9)	0.500	48.4	1.55E-02 (1.5E-04)	2.263 (2E-03)	0.2035 (1.2E-04)
	4:12	67.9 (0.8)	0.500	48.4	1.57E-02 (1.5E-04)	2.264 (2E-03)	0.2035 (1.2E-04)
75	4:28	72.8 (1.0)	0.499	48.5	1.61E-02 (1.6E-04)	2.261 (2E-03)	0.2031 (1.2E-04)
	4:39	72.7 (0.9)	0.499	48.5	1.62E-02 (1.6E-04)	2.262 (2E-03)	0.2031 (1.2E-04)
	4:49	72.8 (0.9)	0.499	48.5	1.62E-02 (1.6E-04)	2.262 (2E-03)	0.2031 (1.2E-04)
80	5:12	77.7 (1.1)	0.499	48.6	1.67E-02 (1.6E-04)	2.262 (2E-03)	0.2026 (1.1E-04)
	5:20	77.8 (1.1)	0.499	48.7	1.67E-02 (1.6E-04)	2.263 (2E-03)	0.2025 (1.2E-04)
	5:25	77.9 (1.1)	0.499	48.7	1.68E-02 (1.6E-04)	2.263 (2E-03)	0.2025 (1.2E-04)
	5:35	78.3 (1.1)	0.499	48.7	1.69E-02 (1.6E-04)	2.264 (2E-03)	0.2025 (1.1E-04)
83	5:52	81.1 (1.1)	0.499	48.8	1.70E-02 (1.6E-04)	2.263 (2E-03)	0.2021 (1.2E-04)
	6:02	81.1 (1.1)	0.499	48.7	1.71E-02 (1.6E-04)	2.264 (2E-03)	0.2021 (1.2E-04)
	6:12	81.0 (1.1)	0.499	48.7	1.71E-02 (1.6E-04)	2.264 (2E-03)	0.2021 (1.2E-04)
86	6:28	83.9 (1.2)	0.499	48.8	1.74E-02 (1.6E-04)	2.265 (2E-03)	0.2018 (1.1E-04)
	6:37	83.7 (1.2)	0.499	48.8	1.74E-02 (1.6E-04)	2.265 (2E-03)	0.2017 (1.1E-04)
	6:48	83.6 (1.2)	0.499	48.8	1.75E-02 (1.6E-04)	2.266 (2E-03)	0.2018 (1.1E-04)
89	7:04	86.5 (1.2)	0.499	48.9	1.78E-02 (1.6E-04)	2.267 (2E-03)	0.2015 (1.1E-04)
	7:14	86.5 (1.2)	0.499	48.9	1.79E-02 (1.6E-04)	2.267 (2E-03)	0.2015 (1.1E-04)
	7:24	86.4 (1.2)	0.499	48.9	1.79E-02 (1.6E-04)	2.267 (2E-03)	0.2015 (1.1E-04)
	7:34	86.4 (1.2)	0.499	48.9	1.79E-02 (1.6E-04)	2.268 (2E-03)	0.2015 (1.1E-04)

Table S3 (Continued).

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (±) [°C]	f -	N [sites chain ⁻¹]	C (±) [cm ⁻¹]	R_g (±) [nm]	χ_{eff} (±) -
91	7:49	88.4 (1.3)	0.499	49.0	1.81E-02 (1.6E-04)	2.268 (2E-03)	0.2013 (1.1E-04)
	7:59	88.5 (1.3)	0.499	49.0	1.82E-02 (1.6E-04)	2.269 (2E-03)	0.2013 (1.1E-04)
	8:09	88.6 (1.3)	0.499	49.0	1.82E-02 (1.6E-04)	2.269 (2E-03)	0.2013 (1.1E-04)
	8:19	88.7 (1.3)	0.499	49.0	1.83E-02 (1.6E-04)	2.269 (2E-03)	0.2013 (1.1E-04)
93	8:36	90.7 (1.3)	0.499	49.0	1.85E-02 (1.6E-04)	2.270 (2E-03)	0.2011 (1.1E-04)
	8:46	90.7 (1.3)	0.499	49.0	1.84E-02 (1.6E-04)	2.270 (2E-03)	0.2012 (1.1E-04)
	8:56	90.7 (1.3)	0.499	49.0	1.85E-02 (1.6E-04)	2.271 (2E-03)	0.2012 (1.1E-04)
95	9:31	92.8 (1.4)	0.499	49.1	1.89E-02 (1.6E-04)	2.274 (2E-03)	0.2014 (9.9E-05)
	9:42	92.7 (1.4)	0.499	49.1	1.89E-02 (1.6E-04)	2.275 (2E-03)	0.2015 (9.8E-05)
	9:51	92.9 (1.4)	0.499	49.1	1.90E-02 (1.6E-04)	2.276 (2E-03)	0.2016 (9.5E-05)
96	10:06	93.8 (1.4)	0.499	49.1	1.94E-02 (1.5E-04)	2.279 (2E-03)	0.2021 (8.6E-05)
	10:16	93.8 (1.4)	0.499	49.1	1.95E-02 (1.5E-04)	2.281 (2E-03)	0.2023 (8.3E-05)
	10:26	93.6 (1.4)	0.499	49.1	1.95E-02 (1.5E-04)	2.282 (2E-03)	0.2023 (8.3E-05)
97	10:44	94.5 (1.4)	0.499	49.2	2.00E-02 (1.5E-04)	2.285 (2E-03)	0.2028 (7.4E-05)
	10:54	94.5 (1.4)	0.499	49.2	2.00E-02 (1.5E-04)	2.286 (2E-03)	0.2029 (7.2E-05)
	11:04	94.4 (1.4)	0.499	49.2	2.02E-02 (1.5E-04)	2.287 (2E-03)	0.2030 (7.0E-05)
	11:14	94.3 (1.4)	0.499	49.2	2.03E-02 (1.5E-04)	2.288 (2E-03)	0.2031 (6.9E-05)
	11:24	94.3 (1.4)	0.499	49.2	2.04E-02 (1.5E-04)	2.288 (1E-03)	0.2032 (6.8E-05)
	11:32	94.3 (1.4)	0.499	49.2	2.04E-02 (1.5E-04)	2.289 (1E-03)	0.2033 (6.7E-05)
98	11:48	95.3 (1.4)	0.499	49.2	2.12E-02 (1.4E-04)	2.292 (1E-03)	0.2037 (5.9E-05)
	11:58	95.3 (1.4)	0.499	49.2	2.14E-02 (1.4E-04)	2.294 (1E-03)	0.2038 (5.7E-05)
	12:08	95.3 (1.4)	0.499	49.2	2.16E-02 (1.4E-04)	2.294 (1E-03)	0.2039 (5.5E-05)
	12:18	95.3 (1.4)	0.499	49.2	2.18E-02 (1.4E-04)	2.295 (1E-03)	0.2040 (5.4E-05)
	12:29	95.3 (1.4)	0.499	49.2	2.19E-02 (1.4E-04)	2.296 (1E-03)	0.2041 (5.3E-05)
	12:38	95.3 (1.4)	0.499	49.2	2.21E-02 (1.4E-04)	2.297 (1E-03)	0.2041 (5.2E-05)
99	12:54	96.3 (1.4)	0.499	49.2	2.29E-02 (1.4E-04)	2.299 (1E-03)	0.2044 (4.7E-05)
	13:04	96.2 (1.4)	0.499	49.2	2.32E-02 (1.4E-04)	2.300 (1E-03)	0.2044 (4.6E-05)
	13:14	96.2 (1.4)	0.499	49.2	2.34E-02 (1.4E-04)	2.300 (1E-03)	0.2045 (4.5E-05)
	13:24	96.3 (1.4)	0.499	49.2	2.36E-02 (1.4E-04)	2.301 (1E-03)	0.2046 (4.4E-05)
	13:34	96.2 (1.4)	0.499	49.2	2.37E-02 (1.4E-04)	2.301 (1E-03)	0.2046 (4.4E-05)
	13:45	96.2 (1.4)	0.499	49.2	2.39E-02 (1.4E-04)	2.302 (1E-03)	0.2047 (4.3E-05)
100	14:01	97.2 (1.5)	0.499	49.2	2.49E-02 (1.4E-04)	2.303 (1E-03)	0.2048 (3.9E-05)
	14:11	97.2 (1.5)	0.499	49.2	2.51E-02 (1.4E-04)	2.304 (1E-03)	0.2048 (3.9E-05)
	14:21	97.2 (1.5)	0.499	49.2	2.54E-02 (1.4E-04)	2.304 (1E-03)	0.2049 (3.8E-05)
	14:32	97.2 (1.5)	0.499	49.2	2.56E-02 (1.4E-04)	2.305 (1E-03)	0.2049 (3.8E-05)
	14:42	97.2 (1.5)	0.499	49.2	2.57E-02 (1.4E-04)	2.305 (1E-03)	0.2049 (3.7E-05)
	14:52	97.2 (1.5)	0.499	49.2	2.58E-02 (1.4E-04)	2.306 (1E-03)	0.2049 (3.7E-05)

Table S3 (Continued).

T_{set} [°C]	Time _{meas} [hr:min]	T_{meas} (±) [°C]	f -	N [sites chain ⁻¹]	C (±) [cm ⁻¹]	R_g (±) [nm]	χ_{eff} (±) -
101	15:23	98.2 (1.5)	0.499	49.3	2.70E-02 (1.4E-04)	2.306 (1E-03)	0.2050 (3.5E-05)
	15:33	98.1 (1.5)	0.499	49.3	2.71E-02 (1.4E-04)	2.306 (1E-03)	0.2050 (3.4E-05)
	15:41	98.1 (1.5)	0.499	49.3	2.71E-02 (1.4E-04)	2.307 (1E-03)	0.2050 (3.4E-05)
	15:51	98.2 (1.5)	0.499	49.3	2.73E-02 (1.4E-04)	2.307 (1E-03)	0.2050 (3.4E-05)
	15:58	98.2 (1.5)	0.499	49.3	2.74E-02 (1.4E-04)	2.308 (1E-03)	0.2050 (3.4E-05)
102	16:14	99.2 (1.5)	0.499	49.3	2.83E-02 (1.4E-04)	2.308 (1E-03)	0.2050 (3.2E-05)
	16:24	99.2 (1.5)	0.499	49.3	2.84E-02 (1.4E-04)	2.308 (1E-03)	0.2051 (3.2E-05)
	16:34	99.2 (1.5)	0.499	49.3	2.86E-02 (1.4E-04)	2.309 (1E-03)	0.2051 (3.1E-05)
	16:44	99.2 (1.5)	0.499	49.3	2.87E-02 (1.4E-04)	2.309 (1E-03)	0.2051 (3.1E-05)
	16:54	99.3 (1.5)	0.499	49.3	2.88E-02 (1.4E-04)	2.309 (1E-03)	0.2051 (3.1E-05)
	17:04	99.3 (1.5)	0.499	49.3	2.89E-02 (1.4E-04)	2.310 (1E-03)	0.2051 (3.1E-05)
103	17:21	100.3 (1.5)	0.499	49.3	2.97E-02 (1.5E-04)	2.310 (1E-03)	0.2050 (3.0E-05)
	17:31	100.3 (1.5)	0.499	49.3	2.98E-02 (1.5E-04)	2.310 (1E-03)	0.2051 (3.0E-05)
	17:41	100.4 (1.5)	0.499	49.3	3.00E-02 (1.5E-04)	2.310 (1E-03)	0.2051 (3.0E-05)
	17:51	100.3 (1.5)	0.499	49.3	3.00E-02 (1.5E-04)	2.311 (1E-03)	0.2051 (3.0E-05)
	18:01	100.3 (1.5)	0.499	49.3	3.01E-02 (1.5E-04)	2.311 (1E-03)	0.2051 (3.0E-05)
104	18:27	101.3 (1.6)	0.499	49.4	3.04E-02 (1.5E-04)	2.311 (1E-03)	0.2050 (2.9E-05)
	18:37	101.2 (1.6)	0.499	49.4	3.04E-02 (1.5E-04)	2.311 (1E-03)	0.2050 (2.9E-05)
	18:47	101.2 (1.6)	0.499	49.4	3.04E-02 (1.5E-04)	2.311 (1E-03)	0.2050 (2.9E-05)
	18:57	101.2 (1.6)	0.499	49.4	3.05E-02 (1.5E-04)	2.311 (1E-03)	0.2050 (2.9E-05)
	19:07	101.1 (1.6)	0.499	49.4	3.05E-02 (1.5E-04)	2.312 (1E-03)	0.2050 (2.9E-05)
	19:17	101.2 (1.6)	0.499	49.4	3.06E-02 (1.5E-04)	2.312 (1E-03)	0.2050 (2.9E-05)
105	19:32	102.1 (1.6)	0.499	49.4	3.09E-02 (1.5E-04)	2.312 (1E-03)	0.2049 (2.9E-05)
	19:42	102.1 (1.6)	0.499	49.4	3.11E-02 (1.5E-04)	2.312 (1E-03)	0.2049 (2.8E-05)
	19:52	102.0 (1.6)	0.499	49.4	3.11E-02 (1.5E-04)	2.313 (1E-03)	0.2049 (2.8E-05)
	20:02	102.0 (1.6)	0.499	49.4	3.12E-02 (1.5E-04)	2.313 (1E-03)	0.2049 (2.8E-05)
	20:03	101.9 (1.6)	0.499	49.4	3.10E-02 (1.5E-04)	2.313 (1E-03)	0.2049 (2.8E-05)
	20:13	102.0 (1.6)	0.499	49.4	3.11E-02 (1.5E-04)	2.313 (1E-03)	0.2049 (2.8E-05)
106	20:30	102.9 (1.6)	0.499	49.4	3.14E-02 (1.5E-04)	2.313 (1E-03)	0.2048 (2.8E-05)
	20:40	102.9 (1.6)	0.499	49.4	3.13E-02 (1.5E-04)	2.313 (1E-03)	0.2048 (2.8E-05)
	20:50	102.8 (1.6)	0.499	49.4	3.15E-02 (1.5E-04)	2.314 (1E-03)	0.2048 (2.8E-05)
	21:17	102.9 (1.6)	0.499	49.4	3.16E-02 (1.5E-04)	2.314 (1E-03)	0.2048 (2.8E-05)
107	21:43	103.8 (1.6)	0.499	49.4	3.17E-02 (1.5E-04)	2.314 (1E-03)	0.2047 (2.8E-05)
	21:53	103.8 (1.6)	0.499	49.4	3.17E-02 (1.5E-04)	2.314 (1E-03)	0.2047 (2.7E-05)
	22:03	103.8 (1.6)	0.499	49.4	3.17E-02 (1.5E-04)	2.314 (1E-03)	0.2047 (2.7E-05)
	22:13	103.8 (1.6)	0.499	49.4	3.17E-02 (1.5E-04)	2.314 (1E-03)	0.2047 (2.7E-05)
	22:23	103.8 (1.6)	0.499	49.4	3.17E-02 (1.5E-04)	2.314 (1E-03)	0.2047 (2.7E-05)
	22:31	103.9 (1.6)	0.499	49.4	3.17E-02 (1.5E-04)	2.314 (1E-03)	0.2047 (2.7E-05)

Table S3 (Continued).

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (\pm) [°C]	f -	N [sites chain ⁻¹]	C (\pm) [cm ⁻¹]	R_g (\pm) [nm]	χ_{eff} (\pm) -
110	23:00	106.7 (1.7)	0.499	49.5	3.20E-02 (1.5E-04)	2.311 (1E-03)	0.2043 (2.4E-05)
	23:10	106.8 (1.7)	0.499	49.5	3.19E-02 (1.5E-04)	2.311 (1E-03)	0.2043 (2.4E-05)
	23:19	106.7 (1.7)	0.499	49.5	3.18E-02 (1.5E-04)	2.311 (1E-03)	0.2043 (2.4E-05)
115	23:40	111.6 (1.8)	0.499	49.7	3.18E-02 (1.5E-04)	2.307 (1E-03)	0.2037 (2.4E-05)
	23:50	111.6 (1.8)	0.499	49.7	3.16E-02 (1.4E-04)	2.306 (1E-03)	0.2037 (2.4E-05)
	24:00	111.8 (1.8)	0.499	49.7	3.15E-02 (1.4E-04)	2.304 (1E-03)	0.2037 (2.4E-05)
120	24:18	116.6 (2.0)	0.499	49.8	3.15E-02 (1.5E-04)	2.300 (1E-03)	0.2030 (2.4E-05)
	24:29	116.9 (1.9)	0.499	49.9	3.12E-02 (1.4E-04)	2.298 (1E-03)	0.2030 (2.4E-05)
	24:39	116.4 (1.9)	0.499	49.8	3.13E-02 (1.4E-04)	2.298 (1E-03)	0.2031 (2.4E-05)
125	24:57	120.9 (2.1)	0.499	50.0	3.14E-02 (1.5E-04)	2.294 (1E-03)	0.2024 (2.5E-05)
	25:07	121.5 (2.1)	0.499	50.0	3.10E-02 (1.5E-04)	2.293 (1E-03)	0.2023 (2.5E-05)
	25:17	121.3 (2.0)	0.499	50.0	3.11E-02 (1.5E-04)	2.292 (1E-03)	0.2024 (2.5E-05)
130	25:34	126.2 (2.2)	0.499	50.1	3.11E-02 (1.5E-04)	2.288 (1E-03)	0.2017 (2.5E-05)
	25:45	126.8 (2.2)	0.499	50.2	3.10E-02 (1.5E-04)	2.286 (1E-03)	0.2016 (2.5E-05)
	25:54	126.0 (2.2)	0.499	50.1	3.10E-02 (1.5E-04)	2.286 (1E-03)	0.2017 (2.5E-05)
135	26:10	130.9 (2.3)	0.499	50.3	3.10E-02 (1.5E-04)	2.282 (1E-03)	0.2010 (2.6E-05)
	26:21	131.3 (2.3)	0.499	50.3	3.08E-02 (1.5E-04)	2.281 (1E-03)	0.2009 (2.6E-05)
	26:31	131.8 (2.3)	0.499	50.3	3.08E-02 (1.5E-04)	2.280 (1E-03)	0.2009 (2.6E-05)
140	26:47	136.4 (2.5)	0.499	50.5	3.10E-02 (1.5E-04)	2.277 (1E-03)	0.2002 (2.7E-05)
	26:58	136.4 (2.5)	0.499	50.5	3.08E-02 (1.5E-04)	2.277 (1E-03)	0.2002 (2.7E-05)
	27:07	136.6 (2.5)	0.499	50.5	3.09E-02 (1.5E-04)	2.277 (1E-03)	0.2002 (2.7E-05)
145	27:23	141.3 (2.6)	0.499	50.6	3.10E-02 (1.6E-04)	2.274 (1E-03)	0.1995 (2.8E-05)
	27:34	141.6 (2.6)	0.499	50.6	3.07E-02 (1.5E-04)	2.273 (1E-03)	0.1994 (2.8E-05)
	27:43	142.0 (2.6)	0.499	50.6	3.08E-02 (1.6E-04)	2.273 (1E-03)	0.1994 (2.8E-05)

Table S4. Sample 1 fit parameters for I_{bdg} at all measured temperatures. T_{set} represents the controller setpoint, $Time_{meas}$ is the cumulative experimental time since the first scattering measurement, T_{meas} is the experimentally measured temperature, and y_1, y_2 correspond to the constants in Equation 13 of the main text. All reported

errors represent one standard deviation of the fitting error.

T_{set} [°C]	$Time_{meas}$ [hr:min]	T_{meas} (±) [°C]	y_1 (±) [cm ⁻¹]	y_2 (±) [nm ⁻¹]
30	0:00	28.9 (0.1)	3.57E-02 (3.E-04)	0.36 (0.02)
40	0:42	38.6 (0.3)	7.23E-02 (3.E-04)	0.33 (0.01)
	0:52	38.6 (0.3)	7.18E-02 (3.E-04)	0.32 (0.01)
	1:05	38.6 (0.3)	7.19E-02 (3.E-04)	0.32 (0.01)
50	1:21	48.3 (0.5)	7.30E-02 (3.E-04)	0.33 (0.01)
	1:31	48.3 (0.5)	7.26E-02 (3.E-04)	0.33 (0.01)
	1:44	48.3 (0.5)	7.38E-02 (3.E-04)	0.32 (0.01)
55	1:59	53.2 (0.6)	3.80E-02 (3.E-04)	0.37 (0.02)
	2:09	53.2 (0.6)	3.72E-02 (3.E-04)	0.36 (0.02)
	2:18	53.2 (0.5)	3.68E-02 (3.E-04)	0.35 (0.02)
60	2:35	58.1 (0.7)	3.71E-02 (3.E-04)	0.36 (0.02)
	2:44	58.1 (0.7)	3.66E-02 (3.E-04)	0.36 (0.02)
	2:54	58.2 (0.7)	3.65E-02 (3.E-04)	0.36 (0.02)
65	3:15	63.0 (0.8)	3.71E-02 (3.E-04)	0.37 (0.02)
	3:24	63.0 (0.7)	3.70E-02 (3.E-04)	0.38 (0.02)
	3:37	63.0 (0.7)	3.74E-02 (3.E-04)	0.39 (0.01)
70	3:52	67.9 (0.9)	3.77E-02 (3.E-04)	0.39 (0.01)
	4:02	67.9 (0.9)	3.80E-02 (3.E-04)	0.40 (0.01)
	4:12	67.9 (0.8)	3.88E-02 (3.E-04)	0.42 (0.01)
75	4:28	72.8 (1.0)	3.94E-02 (3.E-04)	0.38 (0.02)
	4:39	72.7 (0.9)	3.97E-02 (3.E-04)	0.39 (0.01)
	4:49	72.8 (0.9)	3.95E-02 (3.E-04)	0.39 (0.01)
80	5:12	77.7 (1.1)	4.04E-02 (3.E-04)	0.40 (0.01)
	5:20	77.8 (1.1)	3.99E-02 (3.E-04)	0.40 (0.01)
	5:25	77.9 (1.1)	3.99E-02 (3.E-04)	0.41 (0.01)
	5:35	78.3 (1.1)	4.04E-02 (3.E-04)	0.42 (0.01)
83	5:52	81.1 (1.1)	4.19E-02 (3.E-04)	0.40 (0.01)
	6:02	81.1 (1.1)	4.19E-02 (3.E-04)	0.41 (0.01)
	6:12	81.0 (1.1)	4.17E-02 (3.E-04)	0.41 (0.01)
86	6:28	83.9 (1.2)	4.23E-02 (3.E-04)	0.42 (0.01)
	6:37	83.7 (1.2)	4.16E-02 (3.E-04)	0.41 (0.01)
	6:48	83.6 (1.2)	4.18E-02 (3.E-04)	0.42 (0.01)
89	7:04	86.5 (1.2)	4.23E-02 (3.E-04)	0.43 (0.01)
	7:14	86.5 (1.2)	4.22E-02 (3.E-04)	0.43 (0.01)
	7:24	86.4 (1.2)	4.21E-02 (3.E-04)	0.43 (0.01)
	7:34	86.4 (1.2)	4.23E-02 (3.E-04)	0.44 (0.01)

Table S4 (Continued).

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (\pm) [°C]	y_1 (\pm) [cm ⁻¹]	y_2 (\pm) [nm ⁻¹]
91	7:49	88.4 (1.3)	4.24E-02 (3.E-04)	0.44 (0.01)
	7:59	88.5 (1.3)	4.22E-02 (3.E-04)	0.44 (0.01)
	8:09	88.6 (1.3)	4.23E-02 (3.E-04)	0.45 (0.01)
	8:19	88.7 (1.3)	4.24E-02 (3.E-04)	0.45 (0.01)
93	8:36	90.7 (1.3)	4.22E-02 (3.E-04)	0.46 (0.01)
	8:46	90.7 (1.3)	4.19E-02 (3.E-04)	0.46 (0.01)
	8:56	90.7 (1.3)	4.20E-02 (3.E-04)	0.46 (0.01)
95	9:31	92.8 (1.4)	4.24E-02 (3.E-04)	0.46 (0.01)
	9:42	92.7 (1.4)	4.24E-02 (3.E-04)	0.46 (0.01)
	9:51	92.9 (1.4)	4.24E-02 (3.E-04)	0.47 (0.01)
96	10:06	93.8 (1.4)	4.25E-02 (3.E-04)	0.47 (0.01)
	10:16	93.8 (1.4)	4.24E-02 (3.E-04)	0.47 (0.01)
	10:26	93.6 (1.4)	4.22E-02 (3.E-04)	0.47 (0.01)
97	10:44	94.5 (1.4)	4.24E-02 (3.E-04)	0.48 (0.01)
	10:54	94.5 (1.4)	4.18E-02 (3.E-04)	0.48 (0.01)
	11:04	94.4 (1.4)	4.20E-02 (3.E-04)	0.48 (0.01)
	11:14	94.3 (1.4)	4.21E-02 (3.E-04)	0.49 (0.01)
	11:24	94.3 (1.4)	4.20E-02 (3.E-04)	0.49 (0.01)
	11:32	94.3 (1.4)	4.17E-02 (3.E-04)	0.49 (0.01)
98	11:48	95.3 (1.4)	4.14E-02 (3.E-04)	0.50 (0.01)
	11:58	95.3 (1.4)	4.10E-02 (3.E-04)	0.50 (0.01)
	12:08	95.3 (1.4)	4.09E-02 (3.E-04)	0.50 (0.01)
	12:18	95.3 (1.4)	4.10E-02 (3.E-04)	0.50 (0.01)
	12:29	95.3 (1.4)	4.09E-02 (3.E-04)	0.51 (0.01)
	12:38	95.3 (1.4)	4.06E-02 (3.E-04)	0.51 (0.01)
99	12:54	96.3 (1.4)	4.00E-02 (3.E-04)	0.51 (0.01)
	13:04	96.2 (1.4)	3.99E-02 (3.E-04)	0.51 (0.01)
	13:14	96.2 (1.4)	3.96E-02 (3.E-04)	0.51 (0.01)
	13:24	96.3 (1.4)	3.95E-02 (3.E-04)	0.52 (0.01)
	13:34	96.2 (1.4)	3.91E-02 (3.E-04)	0.52 (0.01)
	13:45	96.2 (1.4)	3.90E-02 (3.E-04)	0.52 (0.01)
100	14:01	97.2 (1.5)	3.82E-02 (3.E-04)	0.52 (0.01)
	14:11	97.2 (1.5)	3.80E-02 (3.E-04)	0.53 (0.01)
	14:21	97.2 (1.5)	3.80E-02 (3.E-04)	0.53 (0.01)
	14:32	97.2 (1.5)	3.78E-02 (3.E-04)	0.53 (0.01)
	14:42	97.2 (1.5)	3.77E-02 (3.E-04)	0.53 (0.01)
	14:52	97.2 (1.5)	3.76E-02 (3.E-04)	0.54 (0.01)

Table S4 (Continued).

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (\pm) [°C]	y_1 (\pm) [cm ⁻¹]	y_2 (\pm) [nm ⁻¹]
101	15:23	98.2 (1.5)	3.70E-02 (3.E-04)	0.54 (0.01)
	15:33	98.1 (1.5)	3.65E-02 (3.E-04)	0.54 (0.01)
	15:41	98.1 (1.5)	3.64E-02 (3.E-04)	0.55 (0.01)
	15:51	98.2 (1.5)	3.62E-02 (3.E-04)	0.55 (0.01)
	15:58	98.2 (1.5)	3.63E-02 (3.E-04)	0.55 (0.01)
102	16:14	99.2 (1.5)	3.57E-02 (3.E-04)	0.55 (0.01)
	16:24	99.2 (1.5)	3.53E-02 (3.E-04)	0.56 (0.01)
	16:34	99.2 (1.5)	3.51E-02 (3.E-04)	0.56 (0.01)
	16:44	99.2 (1.5)	3.49E-02 (3.E-04)	0.56 (0.01)
	16:54	99.3 (1.5)	3.49E-02 (3.E-04)	0.56 (0.01)
	17:04	99.3 (1.5)	3.48E-02 (3.E-04)	0.57 (0.01)
103	17:21	100.3 (1.5)	3.45E-02 (3.E-04)	0.57 (0.01)
	17:31	100.3 (1.5)	3.39E-02 (3.E-04)	0.57 (0.01)
	17:41	100.4 (1.5)	3.38E-02 (3.E-04)	0.58 (0.01)
	17:51	100.3 (1.5)	3.39E-02 (3.E-04)	0.58 (0.01)
	18:01	100.3 (1.5)	3.41E-02 (3.E-04)	0.58 (0.01)
104	18:27	101.3 (1.6)	3.37E-02 (3.E-04)	0.58 (0.01)
	18:37	101.2 (1.6)	3.33E-02 (3.E-04)	0.58 (0.01)
	18:47	101.2 (1.6)	3.32E-02 (3.E-04)	0.59 (0.01)
	18:57	101.2 (1.6)	3.31E-02 (3.E-04)	0.59 (0.01)
	19:07	101.1 (1.6)	3.30E-02 (3.E-04)	0.59 (0.01)
	19:17	101.2 (1.6)	3.30E-02 (3.E-04)	0.59 (0.01)
105	19:32	102.1 (1.6)	3.30E-02 (3.E-04)	0.59 (0.01)
	19:42	102.1 (1.6)	3.27E-02 (3.E-04)	0.60 (0.01)
	19:52	102.0 (1.6)	3.26E-02 (3.E-04)	0.60 (0.01)
	20:02	102.0 (1.6)	3.29E-02 (3.E-04)	0.60 (0.01)
	20:03	101.9 (1.6)	3.26E-02 (3.E-04)	0.60 (0.01)
	20:13	102.0 (1.6)	3.24E-02 (3.E-04)	0.60 (0.01)
106	20:30	102.9 (1.6)	3.25E-02 (3.E-04)	0.60 (0.01)
	20:40	102.9 (1.6)	3.21E-02 (3.E-04)	0.60 (0.01)
	20:50	102.8 (1.6)	3.20E-02 (3.E-04)	0.61 (0.01)
	21:17	102.9 (1.6)	3.20E-02 (3.E-04)	0.61 (0.01)
107	21:43	103.8 (1.6)	3.23E-02 (3.E-04)	0.61 (0.01)
	21:53	103.8 (1.6)	3.18E-02 (3.E-04)	0.62 (0.01)
	22:03	103.8 (1.6)	3.19E-02 (3.E-04)	0.62 (0.01)
	22:13	103.8 (1.6)	3.19E-02 (3.E-04)	0.62 (0.01)
	22:23	103.8 (1.6)	3.19E-02 (3.E-04)	0.62 (0.01)
	22:31	103.9 (1.6)	3.18E-02 (3.E-04)	0.62 (0.01)

Table S4 (Continued).

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (\pm) [°C]	y_1 (\pm) [cm ⁻¹]	y_2 (\pm) [nm ⁻¹]
110	23:00	106.7 (1.7)	3.25E-02 (3.E-04)	0.61 (0.01)
	23:10	106.8 (1.7)	3.26E-02 (3.E-04)	0.61 (0.01)
	23:19	106.7 (1.7)	3.25E-02 (3.E-04)	0.60 (0.01)
115	23:40	111.6 (1.8)	3.37E-02 (3.E-04)	0.58 (0.01)
	23:50	111.6 (1.8)	3.38E-02 (3.E-04)	0.57 (0.01)
	24:00	111.8 (1.8)	3.43E-02 (3.E-04)	0.56 (0.01)
120	24:18	116.6 (2.0)	3.55E-02 (3.E-04)	0.55 (0.01)
	24:29	116.9 (1.9)	3.54E-02 (3.E-04)	0.53 (0.01)
	24:39	116.4 (1.9)	3.57E-02 (3.E-04)	0.53 (0.01)
125	24:57	120.9 (2.1)	3.71E-02 (3.E-04)	0.52 (0.01)
	25:07	121.5 (2.1)	3.67E-02 (3.E-04)	0.51 (0.01)
	25:17	121.3 (2.0)	3.71E-02 (3.E-04)	0.51 (0.01)
130	25:34	126.2 (2.2)	3.83E-02 (3.E-04)	0.49 (0.01)
	25:45	126.8 (2.2)	3.83E-02 (3.E-04)	0.48 (0.01)
	25:54	126.0 (2.2)	3.86E-02 (3.E-04)	0.47 (0.01)
135	26:10	130.9 (2.3)	4.00E-02 (3.E-04)	0.44 (0.01)
	26:21	131.3 (2.3)	4.00E-02 (4.E-04)	0.42 (0.02)
	26:31	131.8 (2.3)	4.04E-02 (4.E-04)	0.42 (0.02)
140	26:47	136.4 (2.5)	4.16E-02 (4.E-04)	0.42 (0.02)
	26:58	136.4 (2.5)	4.11E-02 (4.E-04)	0.43 (0.02)
	27:07	136.6 (2.5)	4.16E-02 (4.E-04)	0.42 (0.02)
145	27:23	141.3 (2.6)	4.28E-02 (4.E-04)	0.41 (0.02)
	27:34	141.6 (2.6)	4.24E-02 (4.E-04)	0.41 (0.02)
	27:43	142.0 (2.6)	4.28E-02 (4.E-04)	0.40 (0.02)

Table S5. Sample 1 calculated quantities. T_{set} represents the controller setpoint, $\text{Time}_{\text{meas}}$ is the cumulative experimental time since the first scattering measurement, T_{meas} is the experimentally measured temperature, and Q_{ord} , ΔQ , ϕ_{ord} , d_{ord} , d_{dis} correspond to the values in Equations 14-19 of the main text. All reported errors represent the propagation of one standard deviation of the fitting error through the subsequent calculations.

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (±) [°C]	$Q_{\text{ord}} + \Delta Q$ [cm ⁻¹ nm ⁻³]	ϕ_{ord} -	d_{ord} (±) [nm]	d_{dis} (±) [nm]
30	0:00	28.9 (0.1)	1.103	0.735	7.652 (4E-04)	7.496 (7E-03)
40	0:42	38.6 (0.3)	1.091	0.722	7.643 (4E-04)	7.495 (7E-03)
	0:52	38.6 (0.3)	1.079	0.721	7.644 (4E-04)	7.497 (7E-03)
	1:05	38.6 (0.3)	1.082	0.721	7.644 (4E-04)	7.497 (7E-03)
50	1:21	48.3 (0.5)	1.065	0.707	7.633 (4E-04)	7.493 (7E-03)
	1:31	48.3 (0.5)	1.055	0.706	7.634 (4E-04)	7.495 (7E-03)
	1:44	48.3 (0.5)	1.033	0.704	7.630 (4E-04)	7.492 (7E-03)
55	1:59	53.2 (0.6)	1.023	0.697	7.625 (4E-04)	7.486 (7E-03)
	2:09	53.2 (0.6)	1.009	0.697	7.626 (4E-04)	7.486 (7E-03)
	2:18	53.2 (0.5)	1.005	0.697	7.626 (4E-04)	7.487 (7E-03)
60	2:35	58.1 (0.7)	0.989	0.690	7.622 (4E-04)	7.487 (7E-03)
	2:44	58.1 (0.7)	0.979	0.689	7.623 (4E-04)	7.488 (7E-03)
	2:54	58.2 (0.7)	0.978	0.689	7.623 (4E-04)	7.489 (7E-03)
65	3:15	63.0 (0.8)	0.980	0.682	7.619 (4E-04)	7.490 (7E-03)
	3:24	63.0 (0.7)	0.977	0.681	7.620 (4E-04)	7.491 (7E-03)
	3:37	63.0 (0.7)	0.986	0.682	7.621 (4E-04)	7.493 (7E-03)
70	3:52	67.9 (0.9)	0.986	0.674	7.617 (4E-04)	7.492 (7E-03)
	4:02	67.9 (0.9)	0.995	0.673	7.618 (4E-04)	7.495 (7E-03)
	4:12	67.9 (0.8)	1.009	0.673	7.619 (4E-04)	7.498 (7E-03)
75	4:28	72.8 (1.0)	1.036	0.667	7.610 (4E-04)	7.488 (7E-03)
	4:39	72.7 (0.9)	1.041	0.667	7.611 (4E-04)	7.490 (7E-03)
	4:49	72.8 (0.9)	1.042	0.667	7.613 (4E-04)	7.491 (7E-03)
80	5:12	77.7 (1.1)	1.045	0.657	7.608 (4E-04)	7.492 (6E-03)
	5:20	77.8 (1.1)	1.038	0.656	7.609 (4E-04)	7.494 (7E-03)
	5:25	77.9 (1.1)	1.040	0.654	7.609 (4E-04)	7.495 (6E-03)
	5:35	78.3 (1.1)	1.046	0.654	7.610 (4E-04)	7.496 (6E-03)
83	5:52	81.1 (1.1)	1.034	0.646	7.606 (4E-04)	7.495 (7E-03)
	6:02	81.1 (1.1)	1.035	0.646	7.607 (4E-04)	7.497 (7E-03)
	6:12	81.0 (1.1)	1.034	0.646	7.608 (4E-04)	7.498 (7E-03)
86	6:28	83.9 (1.2)	1.039	0.639	7.606 (4E-04)	7.500 (6E-03)
	6:37	83.7 (1.2)	1.030	0.639	7.608 (4E-04)	7.502 (7E-03)
	6:48	83.6 (1.2)	1.035	0.638	7.610 (4E-04)	7.504 (7E-03)
89	7:04	86.5 (1.2)	1.039	0.631	7.607 (5E-04)	7.506 (6E-03)
	7:14	86.5 (1.2)	1.039	0.630	7.608 (5E-04)	7.507 (6E-03)
	7:24	86.4 (1.2)	1.038	0.630	7.609 (5E-04)	7.508 (6E-03)
	7:34	86.4 (1.2)	1.040	0.630	7.610 (5E-04)	7.509 (6E-03)

Table S5 (Continued).

T_{set} [°C]	T_{meas} [hr:min]	T_{meas} (\pm) [°C]	$Q_{\text{ord}} + \Delta Q$ [cm ⁻¹ nm ⁻³]	ϕ_{ord} -	d_{ord} (\pm) [nm]	d_{dis} (\pm) [nm]
91	7:49	88.4 (1.3)	1.041	0.624	7.609 (5E-04)	7.511 (6E-03)
	7:59	88.5 (1.3)	1.040	0.623	7.610 (5E-04)	7.512 (6E-03)
	8:09	88.6 (1.3)	1.041	0.622	7.610 (5E-04)	7.514 (6E-03)
	8:19	88.7 (1.3)	1.042	0.622	7.611 (5E-04)	7.515 (6E-03)
93	8:36	90.7 (1.3)	1.044	0.615	7.609 (5E-04)	7.517 (6E-03)
	8:46	90.7 (1.3)	1.040	0.614	7.610 (5E-04)	7.517 (6E-03)
	8:56	90.7 (1.3)	1.043	0.613	7.611 (5E-04)	7.520 (6E-03)
95	9:31	92.8 (1.4)	1.047	0.595	7.608 (5E-04)	7.530 (6E-03)
	9:42	92.7 (1.4)	1.048	0.593	7.609 (5E-04)	7.533 (6E-03)
	9:51	92.9 (1.4)	1.049	0.588	7.610 (5E-04)	7.537 (6E-03)
96	10:06	93.8 (1.4)	1.061	0.567	7.609 (5E-04)	7.548 (6E-03)
	10:16	93.8 (1.4)	1.062	0.561	7.610 (5E-04)	7.553 (5E-03)
	10:26	93.6 (1.4)	1.062	0.560	7.611 (5E-04)	7.555 (5E-03)
97	10:44	94.5 (1.4)	1.073	0.534	7.611 (5E-04)	7.566 (5E-03)
	10:54	94.5 (1.4)	1.070	0.527	7.612 (5E-04)	7.570 (5E-03)
	11:04	94.4 (1.4)	1.077	0.523	7.612 (5E-04)	7.573 (5E-03)
	11:14	94.3 (1.4)	1.081	0.519	7.613 (5E-04)	7.575 (5E-03)
	11:24	94.3 (1.4)	1.083	0.514	7.613 (5E-04)	7.578 (5E-03)
	11:32	94.3 (1.4)	1.080	0.509	7.614 (5E-04)	7.581 (5E-03)
98	11:48	95.3 (1.4)	1.093	0.473	7.614 (6E-04)	7.591 (5E-03)
	11:58	95.3 (1.4)	1.090	0.459	7.614 (6E-04)	7.595 (5E-03)
	12:08	95.3 (1.4)	1.095	0.451	7.615 (6E-04)	7.598 (4E-03)
	12:18	95.3 (1.4)	1.098	0.445	7.615 (6E-04)	7.601 (4E-03)
	12:29	95.3 (1.4)	1.098	0.437	7.616 (6E-04)	7.603 (4E-03)
	12:38	95.3 (1.4)	1.099	0.430	7.616 (6E-04)	7.606 (4E-03)
99	12:54	96.3 (1.4)	1.106	0.386	7.616 (7E-04)	7.612 (4E-03)
	13:04	96.2 (1.4)	1.110	0.376	7.616 (7E-04)	7.615 (4E-03)
	13:14	96.2 (1.4)	1.109	0.366	7.617 (7E-04)	7.617 (4E-03)
	13:24	96.3 (1.4)	1.111	0.357	7.617 (7E-04)	7.619 (4E-03)
	13:34	96.2 (1.4)	1.108	0.351	7.617 (7E-04)	7.621 (4E-03)
	13:45	96.2 (1.4)	1.109	0.343	7.618 (7E-04)	7.623 (4E-03)
100	14:01	97.2 (1.5)	1.115	0.294	7.617 (8E-04)	7.627 (4E-03)
	14:11	97.2 (1.5)	1.116	0.284	7.618 (8E-04)	7.629 (4E-03)
	14:21	97.2 (1.5)	1.121	0.276	7.618 (8E-04)	7.631 (4E-03)
	14:32	97.2 (1.5)	1.122	0.268	7.618 (8E-04)	7.633 (4E-03)
	14:42	97.2 (1.5)	1.122	0.262	7.618 (9E-04)	7.634 (4E-03)
	14:52	97.2 (1.5)	1.122	0.257	7.619 (9E-04)	7.636 (4E-03)

Table S5 (Continued).

T_{set} [°C]	T_{meas} [hr:min]	T_{meas} (\pm) [°C]	$Q_{\text{ord}} + \Delta Q$ [cm ⁻¹ nm ⁻³]	ϕ_{ord} -	d_{ord} (\pm) [nm]	d_{dis} (\pm) [nm]
101	15:23	98.2 (1.5)	1.135	0.212	7.617 (1E-03)	7.636 (4E-03)
	15:33	98.1 (1.5)	1.128	0.200	7.618 (1E-03)	7.638 (4E-03)
	15:41	98.1 (1.5)	1.128	0.199	7.618 (1E-03)	7.639 (4E-03)
	15:51	98.2 (1.5)	1.129	0.195	7.618 (1E-03)	7.641 (4E-03)
	15:58	98.2 (1.5)	1.133	0.191	7.618 (1E-03)	7.642 (4E-03)
102	16:14	99.2 (1.5)	1.140	0.153	7.618 (1E-03)	7.643 (4E-03)
	16:24	99.2 (1.5)	1.136	0.145	7.618 (1E-03)	7.644 (4E-03)
	16:34	99.2 (1.5)	1.136	0.135	7.619 (1E-03)	7.646 (4E-03)
	16:44	99.2 (1.5)	1.135	0.133	7.619 (1E-03)	7.647 (4E-03)
	16:54	99.3 (1.5)	1.137	0.131	7.618 (1E-03)	7.648 (4E-03)
	17:04	99.3 (1.5)	1.137	0.126	7.618 (1E-03)	7.649 (4E-03)
103	17:21	100.3 (1.5)	1.141	0.094	7.617 (2E-03)	7.648 (3E-03)
	17:31	100.3 (1.5)	1.137	0.082	7.619 (2E-03)	7.649 (3E-03)
	17:41	100.4 (1.5)	1.137	0.077	7.618 (2E-03)	7.650 (3E-03)
	17:51	100.3 (1.5)	1.138	0.078	7.618 (2E-03)	7.651 (3E-03)
	18:01	100.3 (1.5)	1.141	0.079	7.617 (2E-03)	7.652 (3E-03)
104	18:27	101.3 (1.6)	1.141	0.062	7.617 (2E-03)	7.652 (3E-03)
	18:37	101.2 (1.6)	1.136	0.056	7.619 (2E-03)	7.653 (3E-03)
	18:47	101.2 (1.6)	1.135	0.059	7.617 (2E-03)	7.654 (3E-03)
	18:57	101.2 (1.6)	1.136	0.052	7.619 (2E-03)	7.654 (3E-03)
	19:07	101.1 (1.6)	1.135	0.055	7.617 (2E-03)	7.656 (3E-03)
	19:17	101.2 (1.6)	1.135	0.053	7.618 (2E-03)	7.657 (3E-03)
105	19:32	102.1 (1.6)	1.137	0.040	7.618 (3E-03)	7.656 (3E-03)
	19:42	102.1 (1.6)	1.135	0.030	7.619 (4E-03)	7.657 (3E-03)
	19:52	102.0 (1.6)	1.134	0.028	7.619 (4E-03)	7.658 (3E-03)
	20:02	102.0 (1.6)	1.138	0.032	7.617 (4E-03)	7.659 (3E-03)
	20:03	101.9 (1.6)	1.134	0.033	7.617 (4E-03)	7.659 (3E-03)
	20:13	102.0 (1.6)	1.132	0.032	7.617 (4E-03)	7.660 (3E-03)
106	20:30	102.9 (1.6)	1.134	0.022	7.617 (5E-03)	7.660 (3E-03)
	20:40	102.9 (1.6)	1.129	0.019	7.616 (5E-03)	7.660 (3E-03)
	20:50	102.8 (1.6)	1.130	0.012	7.616 (8E-03)	7.661 (3E-03)
	21:17	102.9 (1.6)	1.133	0.012	7.616 (8E-03)	7.663 (3E-03)
107	21:43	103.8 (1.6)	1.137	0.011	7.616 (8E-03)	7.662 (3E-03)
	21:53	103.8 (1.6)	1.131	0.006	7.617 (1E-02)	7.663 (3E-03)
	22:03	103.8 (1.6)	1.132	0.009	7.614 (9E-03)	7.663 (3E-03)
	22:13	103.8 (1.6)	1.132	0.008	7.614 (1E-02)	7.663 (3E-03)
	22:23	103.8 (1.6)	1.132	0.008	7.614 (1E-02)	7.664 (3E-03)
	22:31	103.9 (1.6)	1.132	0.008	7.614 (1E-02)	7.664 (3E-03)

Table S5 (Continued).

T_{set} [°C]	$\text{Time}_{\text{meas}}$ [hr:min]	T_{meas} (\pm) [°C]	$Q_{\text{ord}} + \Delta Q$ [cm ⁻¹ nm ⁻³]	ϕ_{ord} -	d_{ord} (\pm) [nm]	d_{dis} (\pm) [nm]
110	23:00	106.7 (1.7)	1.138	0.000	- -	7.654 (3E-03)
	23:10	106.8 (1.7)	1.139	0.000	- -	7.653 (3E-03)
	23:19	106.7 (1.7)	1.137	0.000	- -	7.653 (3E-03)
115	23:40	111.6 (1.8)	1.136	0.000	- -	7.640 (3E-03)
	23:50	111.6 (1.8)	1.134	0.000	- -	7.635 (3E-03)
	24:00	111.8 (1.8)	1.139	0.000	- -	7.630 (3E-03)
120	24:18	116.6 (2.0)	1.136	0.000	- -	7.616 (3E-03)
	24:29	116.9 (1.9)	1.129	0.000	- -	7.611 (3E-03)
	24:39	116.4 (1.9)	1.135	0.000	- -	7.609 (3E-03)
125	24:57	120.9 (2.1)	1.134	0.000	- -	7.596 (3E-03)
	25:07	121.5 (2.1)	1.121	0.000	- -	7.592 (3E-03)
	25:17	121.3 (2.0)	1.128	0.000	- -	7.590 (3E-03)
130	25:34	126.2 (2.2)	1.123	0.000	- -	7.576 (3E-03)
	25:45	126.8 (2.2)	1.116	0.000	- -	7.572 (3E-03)
	25:54	126.0 (2.2)	1.121	0.000	- -	7.570 (3E-03)
135	26:10	130.9 (2.3)	1.115	0.000	- -	7.557 (3E-03)
	26:21	131.3 (2.3)	1.109	0.000	- -	7.554 (3E-03)
	26:31	131.8 (2.3)	1.109	0.000	- -	7.552 (3E-03)
140	26:47	136.4 (2.5)	1.109	0.000	- -	7.541 (3E-03)
	26:58	136.4 (2.5)	1.103	0.000	- -	7.540 (3E-03)
	27:07	136.6 (2.5)	1.106	0.000	- -	7.539 (3E-03)
145	27:23	141.3 (2.6)	1.098	0.000	- -	7.529 (4E-03)
	27:34	141.6 (2.6)	1.089	0.000	- -	7.528 (4E-03)
	27:43	142.0 (2.6)	1.092	0.000	- -	7.527 (4E-03)

References

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- (2) Teran, A. A.; Balsara, N. P. Thermodynamics of Block Copolymers with and Without Salt. *J. Phys. Chem. B* **2014**, *118*, 4–17.